Make YInMn Blue Pigment Using Solid State Chemistry

by DrRadium

There is something about blue. All around the world, blue is by far the most popular favorite color. It is also the last color named in every language, as it turns out that people don't name colors until we can make them. For some rather complicated chemical reasons, blue pigments are extremely rare. Most of the blue colors we see, such as the blue color in our eyes and birds feathers are not produced by a pigment, but by light waves interfering with each other. Most plant pigments we think of as blue are actually purple.

Until the dawn of chemical science the best blue came from ground lapis lazuli called ultramarine. While the ancient Egyptians made a synthetic bue, perhap the first synthetic pigment in history, The first new blue from chemistry, Prussian blue was made in 1706, and in 1802 cobalt blue was invented. We figured out how to make synthetic ultramarine in 1826 and by the early 20th century, blue organic pigments such as phthalocyanine blue had been discovered. But with the exception of maganese blue (which is actually cyan), no new inorganic blue pigment was discovered from 1802 until 2009. That year a new blue pigment was serendipitously produced in the lab of Dr. Mas Subramanian at Oregon State University. Andrew Smith, then a grad student, was trying make new materials for electronics from oxides of yttrium, indium, and manganese. The resulting product did not have the desired electronic properties. But it was a bright vibrant blue. From prior experience working at DuPont, Dr. Subramanian knew how rare blue pigments were, and rather than throw it out as a failure as some might have done, they replicated the finding, characterized the product, and investigated the mechanism by which it produced the selective reflectance giving it color. An apparent failure became a whole new line of research for the lab, again confirming that the words most likely said upon a scientific discovery are not 'eureka!' but 'huh, that's odd.'

While technically named indium mangansese yttrium oxide, it was given a common name based on the chemical symbols for elements it is made from: Y (yttrium) In (indium) and Mn (manganese), YInMn blue (pronounced Yin-min) exploded into public consciousness in 2016 after an article about it went viral. It's pure vibrant blue color, remarkable stability, apparent lack of toxicity, and strong near infra-red reflectivity (unlike most blue pigments, it reflects heat rather than absorbs it) made it particularly exciting to many artists and material scientists. Crayola even made a new color crayon (that does not contain any actual YInMn blue) in honor of the discovery. By swapping metal oxides used to include copper, titanium, aluminum, zinc, an iron; a rainbow of other colors can be made: greens, purples, yellows, oranges, and brick reds. Unfortunately few have had the opportunity to use real YInMn blue pigment due to poor availability and a stubbornly high price.

This is unfortunate, because not only is it a remarkable pigment, making it is a remarkable way to learn about solid state chemistry, the method by which it was first made. These are chemical reactions done not in liquid solvents like most introductory chemistry labs teach. but by heating powdered solids together to very high temperatures. Synthesis of YInMn blue is remarkably accessible to even beginning chemistry students. Almost anyone with a kiln that can hold 1200°C (2200°F) and and who can use a scale and a mortar and pestle, can experience an almost magical transformation: mixing black, white, and pale yellow powders together and having it come out of a kiln bright blue.

Solid state synthesis is how many ceramic materials are made. This includes the high temperature superconductor YBCO that is superconductive when cooled only with cheap limitless liquid nitrogen rather than requiring expensive and source limited liquid helium like conventional superconductors. Making YBCO is perhaps the most commonly used solid

state synthesis for teaching purposes, but the process can be fickle with discouraging failures usually only evident after several days of work, and there is no simple visual indicator of success. The synthesis of YInMn blue is much easier and much more reliable than trying to make YBCO and it is clear after the first heating if the process is working. And you don't need liquid nitrogen to show off how cool it is either.

This instructable will describe how to make the newly iconic pigment, YInMn blue. While the synthesis can be carried out by rote to make the pigment without learning much chemistry, it makes a great way to demonstrate solid state chemistry. The only supplies that are essential and have no substitutes are the metal oxides, and a furnace that can heat to 1200°C or greater.

Supplies:

1. *Furnace or kiln capable of reaching 1200°C (about 2200°F) and holding this temperature for several hours.* You must have one if you want there to be any chance of success. Kilns are often rated by 'cone' and you need one that can reach at least cone 6, preferably cone 10.

I used a Rapid-Fire Pro-L made by Tabletop Furnace. They will just reach 1200°C and while heating this hot will shorten the heating element life, I made over 20 full runs (18 hours total per run) before the elements started being questionable. I do suggest using an additional small fan blowing on the electronic controls to help keep them cool if a rapid-fire pro is used. The rapid-fire pro-L runs a little over $600 new.

There is also an instructable on making an electric kiln/furnace by Wolfgar77. It is reported to get to a high enough temperature to make YInMn blue. The furnace it makes is similar in size to the rapid-fire for a fraction of the price. Just use a PID controller, solid state relay (SSR), and a k type thermocouple for the controller. However, I must strongly advise following the suggested changes published for electrical safety.

This kiln, like every other DIY design I have seen, is a bit hazardous as far as electrical safety is concerned. They all rely on people being actively careful around dangerous open 110-230VAC live wires. Probably because it isn't clear to most of us how to connect heating wire up and keep it tucked away without trading a shock hazard for a fire hazard. Following the changes suggested by joppaglass will make it meet commercial kiln safety standards for just a few extra bucks. I also suggest following his advice about pinning the heating elements, though I suggest using 1100-1500 watts of heating to ensure it will get hot enough. Use longer channels for the heating element if needed. He calls the wattage overkill (it is for most uses), but I call it enough to make this project.

2. *Digital scale(s)* You will need a scale that measures to 0.01 gram accuracy (to 10s of milligrams) with at least 50 gram capacity. If you plan to do very small volume reactions under 10 grams total, you should consider a scale accurate to 0.001 grams (milligrams). Small reactions will involve very small amounts of manganese (III) oxide being added. To do this consistently, you need a scale this sensitive. I have both. You can find then on Amazon, eBay, Etsy, and direct from China sites like Ali Express or Banggood. You might consider buying a weight standard if the scale doesn't come with one. Being able to weigh an item known to be 5 grams and seeing your scale show 5.00 or 5.000 grams is very confidence inspiring.

3. *Weighing and powder transfer supplies*. These include paper cups or plastic weigh boats to measure powders into as well as tools such as cheap sets of metal spatulas, palate knives or more formal scientific equipment such as stainless steel scoopulas or microspatulas. As with most scientific supplies these days they can be found on eBay, Etsy, or Amazon. Palate knives may be found at many art stores. Medium weigh boats will be the best size for almost all your needs, though I use a lot of paper dixie cups. I recommend getting some glassine weighting papers as they are one of the few things that manganese (III) oxide will slide off cleanly.

4. *Mortar and Pestle*. I prefer ceramic porcelain models. I also recommend getting one where the end of pestle you hold is flat and not pointed. Having a pointy pestle jabbing into your hand gets old fast. High walled models reduce spills. Add kitchen supply stores online and local to the usual triumvirate of sources (eBay, Etsy, Amazon). Having more than one is nice but not essential. Wash between uses with standard dishwashing detergent and water.

5. *Crucibles*. These hold your pigment while it is heated. Preferably made of fused silica or alumina ceramic. Unglazed porcelain is fine if you can find it. I have also used larger mullite clay crucibles for heating Yttrium oxide to remove water. I

recommend using one crucible only for heating yttrium oxide to remove water and CO_2, another only for making manganese (III) oxide (assuming you cannot find it for sale), and others for making pigment. You will want a large one for dehydrating yttrium oxide and a medium one for the manganese (III) oxide. Unfortunately the sizing on crucibles is often hard to understand. Some are rated in milliliter volumes. But many are listed by weight and usually refer to the weight of a precious metal (often gold) that can be heated. You might think that a 500g crucible would be huge, it isn't. It will hold about 75mL. A 500 to 750g silica crucible or larger is a good size for drying the yttrium oxide. A 100-250g or 30-50mL crucible is good for making manganese (III) oxide, and 100g or 10-30mL crucibles are good for the pigment. Shallow, dish shaped crucibles made of quartz silica are usually the cheapest. But they take up a lot of floor space in the furnace for the volumes they hold. Thinner, taller crucibles cost a little more but are more space efficient. IMPORTANT NOTE: you should NOT coat crucible interiors in flux as you would if you were melting metal. It will contaminate your pigment at best, cause failure at worst. Leave the flux for melting metal and use the crucibles as they are.

6. Metal oxide precursors. Along with the furnace, these have to be correct or you will not be happy with your results. You need yttrium (III) oxide, indium (III) oxide and manganese (III) oxide. These are sometimes listed without the (III) which refers to the ionization state (how charged the metal ions are, in this case +3). You can tell if it is the right oxide by checking the chemical formula. You are looking for Y_2O_3, In_2O_3, and Mn_2O_3. Of the three, you will be able to buy yttrium (III) oxide and indium (III) oxide off eBay or Etsy, and possibly amazon). But you may not find manganese (III) oxide for sale to non-institutional buyers. Don't worry, you can make it from readily sourced magnanese dioxide.

Yttrium (III) oxide (Y_2O_3): I have bought this off eBay. Look for 99.9% or better purity. eBay seller leroy500paul who sells as PaulBrownStudio on Etsy is a reliable source. Many other sellers on eBay are likely reliable as well, I have had mixed luck on Ali Express, several stores had good quality product that worked well, until I got some that was clearly not pure yttrium oxide. Be cautious, but sources from China may be much less expensive as a great deal of the worlds yttrium oxide comes from China.

NOTE: yttrium (III) oxide is hydroscopic. This means it readily absorbs water from the air. Yttrium oxide also absorbs some carbon dioxide from the air to produce yttrium carbonate. The water and carbon dioxide can be removed by heating. Place your yttrium oxide in a clean crucible (you may want to dedicate one for this) and heat at 600°C for 6 hours before use. You can weigh the yttrium oxide before and after heating to see how much water had been absorbed. Allow to cool then store it in a sealed container that is placed inside a larger sealed container with dessicant in it (preferably one that indicates when it has gotten wet and needs to be heated to recharge). This should keep your yttrium (III) oxide dry.

Indium (III) oxide (In_2O_3) can be bought directly from eBay, but the prices vary wildly. Indium oxide is the most expensive component. Expect prices around $2/gram or higher. Although if you get lucky you may find it for a little over $1/gram and buying in much larger amounts from China can yield prices around $0.50/gram. DO NOT GET INDIUM TIN OXIDE (ITO)! It is not what you want! eBay seller leroy500paul who sells as PaulBrownStudio on Etsy is a reliable source of indium oxide and not unreasonable for a small volume seller. eBay seller chemsaversinc1 is also reliable but much more expensive ($3.6 to $8.9 a gram). The least expensive reliable source on eBay was terminal.route-8 but he was based in Kalingrad Russia and is unavailable as of summer 2022 due to trade and financial sanctions resulting from Vladimir Putin's decision to invade Ukraine. If you are interested in buying at least a kilogram, Wuhan Xinrong New Materials (also listed by parent company Wuhan Tuocai Technology) is a very reliable source direct from China with great customer service even for people buying relatively small amounts. I have had excellent, consistent results with their indium (III) oxide.

Manganese (III) oxide (Mn_2O_3) may be difficult to source off eBay or Etsy. If you cannot find it, don't worry! You can easily make it by heating manganese dioxide (MnO_2) to 600°C. Loudwolf brand 99% manganese dioxide is sold by many vendors on eBay, and works very well for this purpose. Simply put some in a crucible and heat to 600°C (no hotter), for 6 hours. This can be done (in a different crucible!) while you are removing water and absorbed carbon dioxide from the yttrium oxide. You can weigh it before and after and see that it looses weight. Weight lost after heating to 200°C is mostly from manganese dioxide loosing any water is has picked up from the air. Weight lost between 200°C and 600°C is likely from oxygen gas produced by the chemical reaction $4MnO_2$ ----> $2Mn_2O_3 + O_2$. Grind the heated product well and store in a sealed container at room temperature.

7. Small airtight containers for pigment and to store oxide precursors, as well as to mix oxide powders by shaking.

NOTE ON SAFETY AND LAB PRACTICES: The synthesis of YInMn blue does not employ chemicals with high toxicity or reactivity and none are caustic or corrosive nor does the process release harmful gasses. Of the three chemicals used, indium oxide and manganese (III) oxide are of the most concern, yttrium oxide is primarily an irritant. Manganese (III) oxide, like other manganese containing chemicals, if chronically ingested is capable of causing a specific toxic syndrome called manganism. It is associated with mood changes, obsessive behaviors and abnormal movements similar to those found in parkinson's disease. Manganism gets noted very frequently whenever manganese is mentioned, sometimes with grave warnings to treat any exposure to manganese as extremely dangerous and likely to cause manganism. This is not the case. The dose makes the poison, and with manganese the frequency too.

Manganism is very rare and found almost entirely in metal workers who breathe manganese metal fumes, in people exposed to manganese through contaminated well water, or in very ill people who have to receive all nutrition through a needle into a vein. Manganese (III) oxide is not well absorbed even if ingested and the amounts used in this experiment are very small compared to the amounts needed to make someone seriously ill. If you decide to increase the challenge and make manganese (III) oxide from molten manganese metal and do so with poor ventilation, you may be exposed to considerably more manganese. Although unless done frequently, it is still a very low risk. *Most importantly, manganism requires chronic exposure to manganese typically day after day, for weeks to months*. A single exposure, especially to the amounts used in this experiment has essentially zero chance of causing manganism.

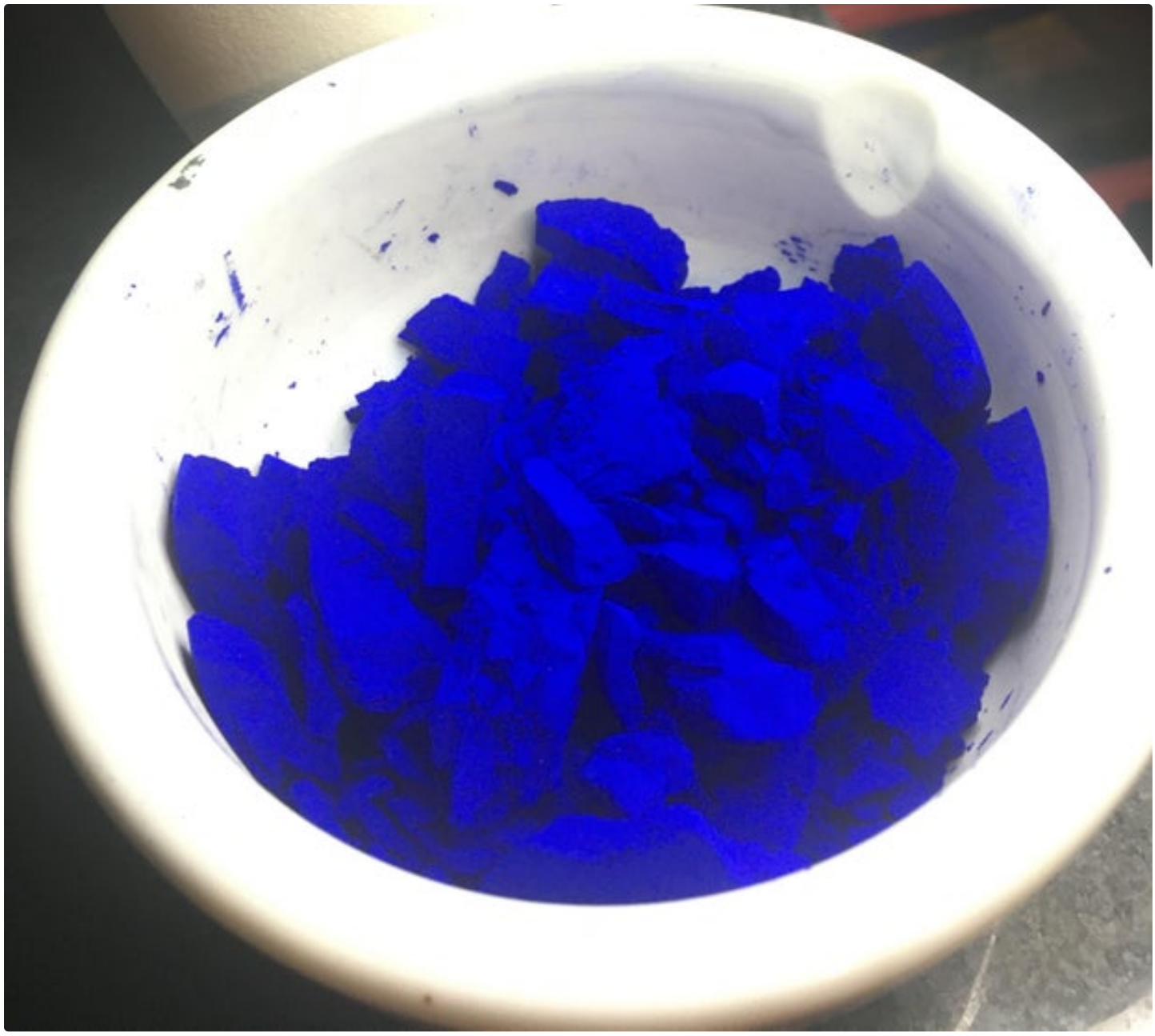
The possible exception is if you have significant prolonged exposure to other sources of manganese and are extremely close to developing it from these exposures. If this is the case and you ingest grams of manganese (III) oxide, it is theoretically possible that this could tip you into having symptoms. So If you drink well water contaminated with high levels of manganese, or are a metal worker exposed to manganese at work, you should exert extra care to ensure you do not ingest by eating or breathing in dust of manganese (III) oxide or the mixed oxides before the first heating. The same is true if you consume manganese regularly through using potassium permanganate as an alternative medicine or if you are a user of the illegal stimulant methcathinone. Ceramic artists frequently doing raku pottery with poor ventilation and who do not use respirators with appropriate filters for metal fumes may also be exposed to high levels of manganese. If these risk factors do not apply to you, you have essentially the same chance of developing manganism as someone who does not perform this experiment.

If these risk factors do apply, this experiment will still add very little to your overall exposure. If you have frequent high level exposure to manganese, and you are concerned you have signs of manganism regardless of where the exposures came from, cease all avoidable exposures and seek medical care immediately as manganism becomes increasingly irreversible as it progresses. Wearing gloves and changing them often as well as using a fitted respirator with P100 or 2297 filters all but eliminates any chance of exposure to any chemicals used here, although it is very likely overkill even for those with strong specific risk factors.

Everyone should follow standard lab safety practices doing this experiment including not eating, drinking, or smoking/vaping while working along with use of standard eye protection. If you accidentally ingest anything, don't panic nothing is likely to happen with small amounts but you should still contact a poison control center immediately to be safe. If chemicals get onto your eyes, flush well with water. Gritty chemicals may cause corneal scratches, if you are having trouble seeing or having severe pain, seek medical care immediately. Wash your hands after working with chemicals and consider using disposable gloves while handling chemicals. Be aware of exposed electrical connections on DIY furnace/kilns. AC electricity from house power lines can cause fatal shocks. The extreme temperatures involved are a fire and burn hazard. Items can be hot enough to cause severe burns while looking the same as if they were cold. Flammable materials near a heated kiln may ignite.

Burn injury and fire are most likely the greatest hazards for this experiment and dwarf the danger from chemical exposure.

Remove anything that can burn from around the furnace/kiln, Avoid looking into the kiln at temperatures above about 900°C for more than a few seconds without eyewear that protects from IR and UV light, such as a #3 (or darker) welders glass or glassworker's didymium glasses.



Step 1: Background on Solid State Chemistry

This section addresses how solid state chemistry differs from more familiar solvent based chemistry. It is mostly for those using this instructable as a STEM exercise or project. Those more interested in the method may want to skip to the next section. Though if you have problems, you may find the solution to your issues here.

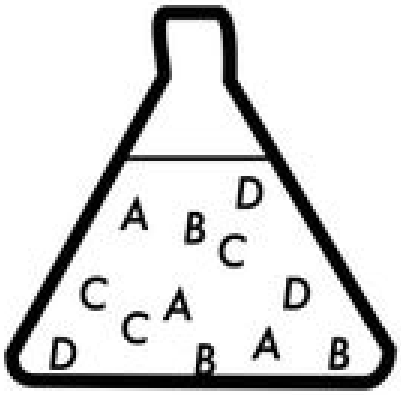
Especially at introductory college level and below, lab coursework tends to center on conventional solvent based chemistry. In these, solids are dissolved in a liquid solvent. This disperses components allowing them to mix and become a homogeneous solution. Because everything is dispersed, dissolved substances are available to react with and will do so assuming the energetics of the reactions are favorable (see top of first figure for this section). In solid state chemistry, there is no solvent and as a result other steps must be taken to mix reactants. This mostly consists of needing to keep particle size small and temperatures very high (see second figure for this section).

In the absence of a solvent, solids only have the capacity to mix where their surfaces meet each other. The zone of mixing becomes larger at higher temperatures as heat is literally motion at the atomic and molecular scale. This motion allows

atoms to migrate more readily, and the zone where mixing takes places grows as the system is hotter. Because the ratio of surface area to volume grows at particle size decrease, small particles have more of their volume within potential mix zones as temperature rises. They also pack together more efficiently as only particles actually in contact will have mixing zones (see third figure for this section).

Differences between solvent based and solid state chemistry flow directly from these differences in mechanism and the their consequences. For example, the elevated temperatures of solid state chemistry due to mixing needs and precludes most organic synthesis because most organic chemicals are destroyed at these temperatures. Or that the lack of a solvent in solid state methods results in making solvent removal unecessary. The nature of inorganic, compared to organic, chemical reactions means less potential for side reactions and thus product purification is often unnecessary. The avoidance of toxic solvents may make the chemistry 'greener' (see bottom of first figure for this section), although the high temperatures needed requires greater power consumption and depending on the source of electricity, may produce produce considerable greenhouse gas emissions.

Solvent-based Chemistry



Solids AB and CD dissolved in a liquid solvent: A, B, C, and D are all dispersed in the solvent and available to react with each other.

Reactions take place readily as components are available to react with each other.

Low temperature reactions allow chemistry with readily destroyed compounds.

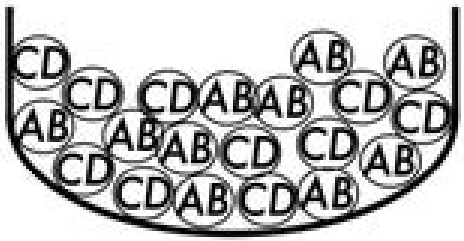
Solids must dissolve in solvent, solvent must not react in unwanted ways.

Many organic solvents are toxic, and can be harmful to the environment. However, use of less toxic solvents and catalysts can be just as 'green.'

Product must be separated from solvent, and organic compounds require purification.

Widely useful for many types of organic and inorganic reactions. Most chemicals are made this way or by reaction of gasses.

Solid State Chemistry



Solids AB and CD are particles of powdered solids packed closely together. Only at very high temperatures will they mix together and react.

Reactions take place only at extremely high temperatures where components of solids can mix with each other.

Many organic molecules are destroyed at these temperatures.

Often very simple reactions: grind together and heat a.k.a. "shake and bake."

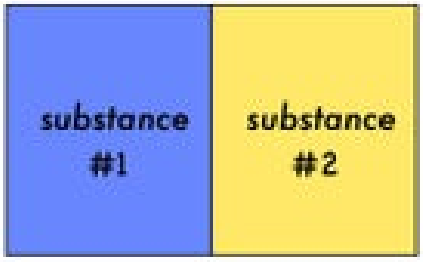
May be 'green' chemistry if reaction does not have toxic gas emisions and source of energy for heating does not emit greenhouse gases.

Product usually requires no separation or purification for use.

Useful for a limited set of inorganic reactions, such as making YBCO superconductor, ceramics, and YInMn blue pigment.

Solids in close direct contact will mix at very high temperatures, making chemical reactions possible

no mixing

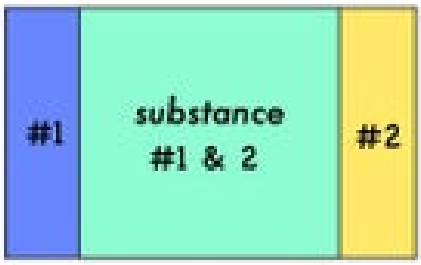


cold

some mixing between substance #1 and #2

hot

almost all of substance #1 and #2 are mixed and may react

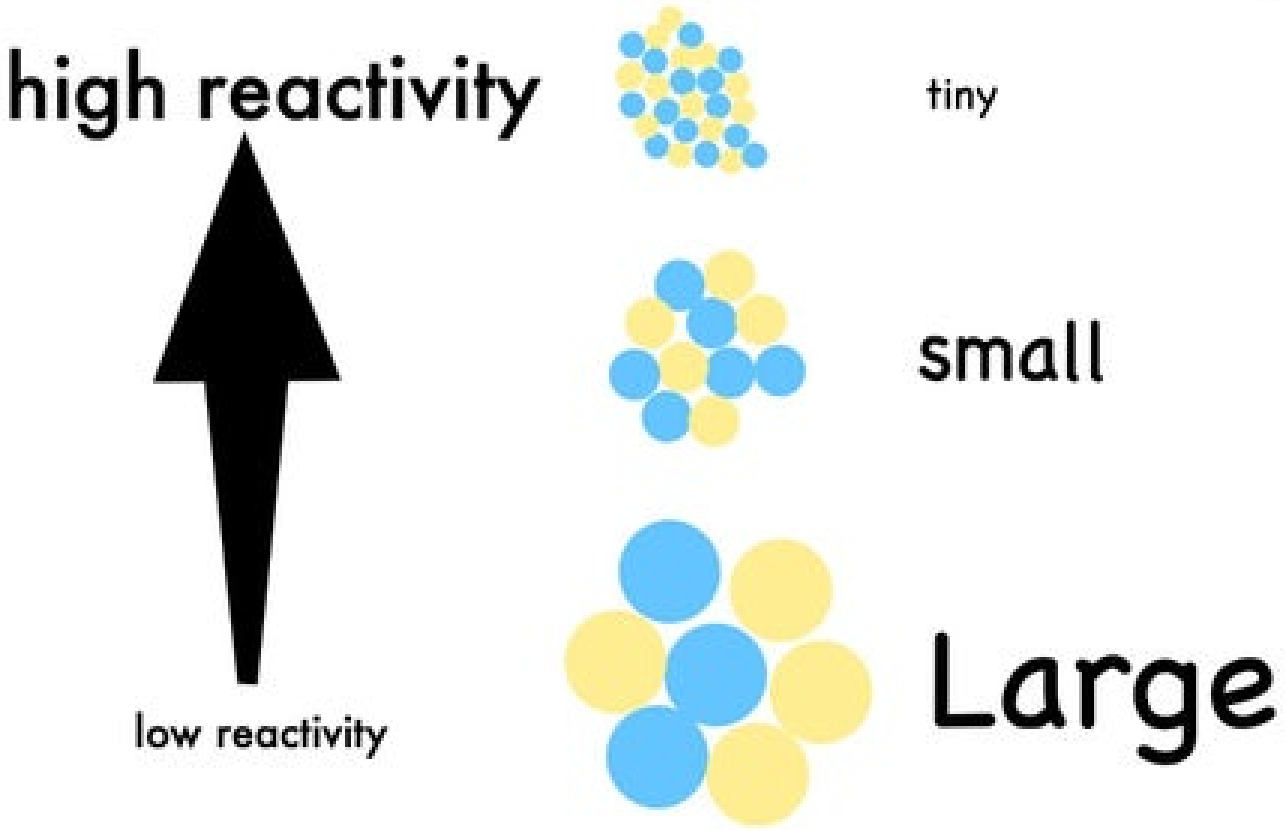


hotter

As substances in a solid state get hotter, the atoms and molecules that they are made of are vibrating faster and faster, allowing some to migrate and even mix with other solid materials. Mixing takes place only at the interface, where the solids are in direct contact with each other. This is the only place that the two substances can chemically react. In general, the hotter the materials are the more atoms and molecules migrate creating larger zones of mixing where chemical reactions can take place. To react, the temperature has to be hot enough for the atoms or molecules in each substance to to mix, and also have the energy to react in the desired way. But it must not be so hot that it undergoes undesired reactions or reaction components boil off.

The reaction of yttrium oxide, indium oxide, and manganese (III) oxide to make YInMn blue pigment requires a temperature of 1200°C. Even a few hundred degrees lower at 1000°C fails because the metal oxides mix but do not react to produce the correct product, and are a pale blue grey.

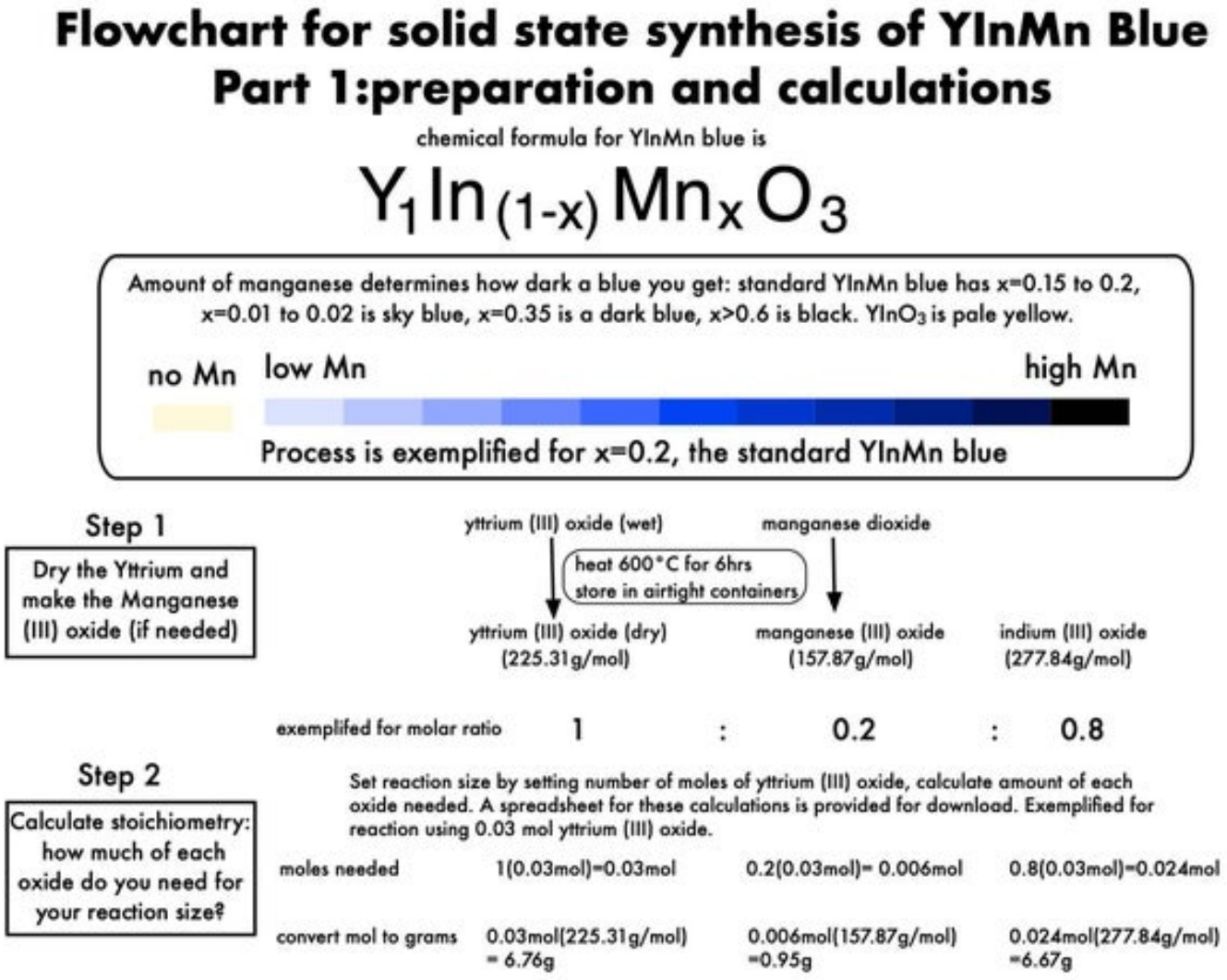
Effect of particle size on solid state reactions

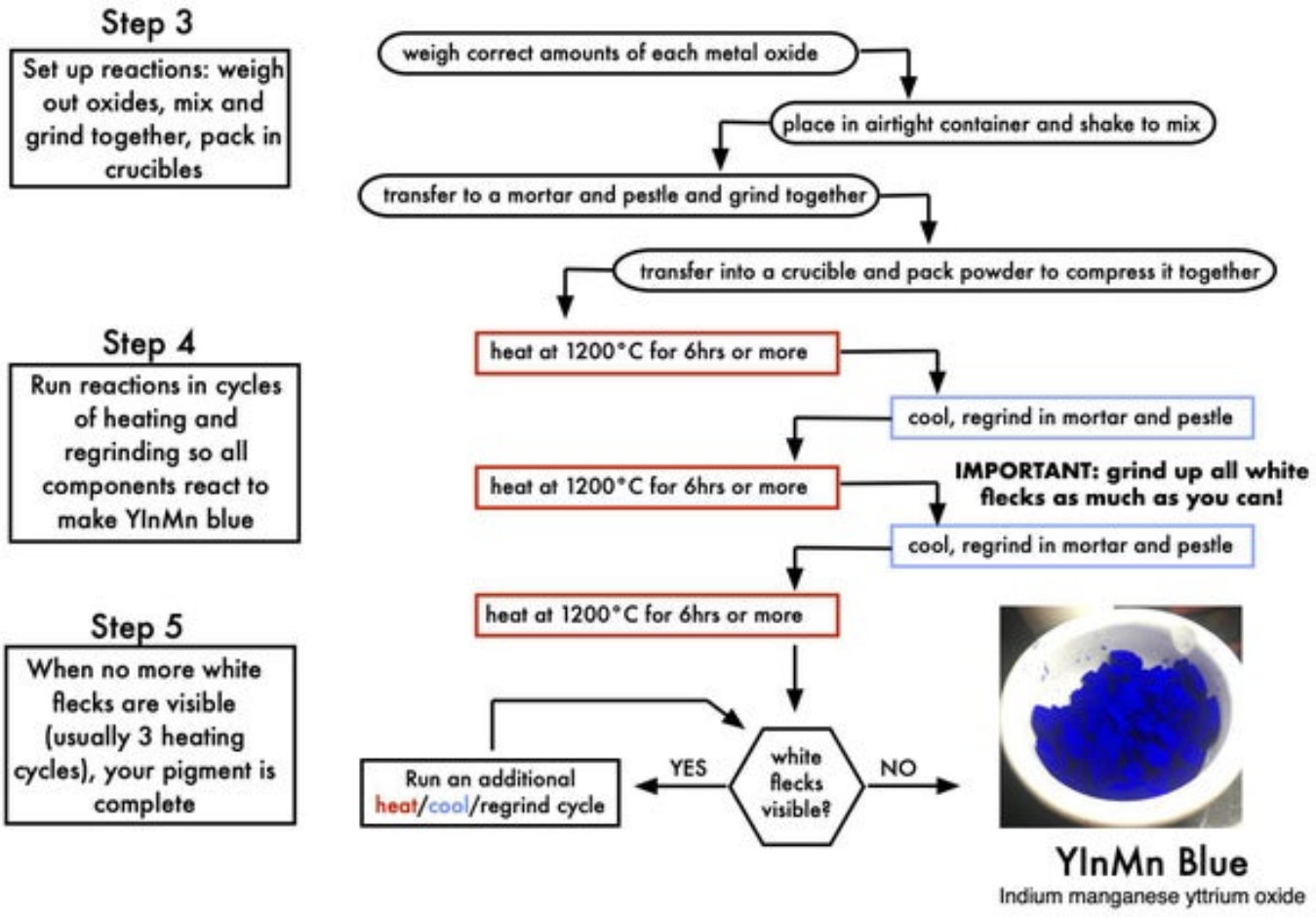


For tiny particles, the entire particle may mix at high temperatures. In large particles, the center of each particle may not mix at all, leaving unreacted material. Also, tiny particles tend to pack together with less space between them. To get small particles ready for making YInMn blue, it is important to mix well by shaking, and grind the mixed metal oxides together in a mortar and pestle, then carefully pack the mixed powder into crucibles. Labs often will use a pellet press to compact the powder into a hard pellet under extremely high pressure, but these are very expensive so we will not be using one. Instead, we will run the reaction several times, grinding the product of each heating cycle together before heating again, 3 or more times to ensure all the metal oxides react.

Step 2: Process Overview Flowcharts

To make the process and work flow for calculations, preparation, and pigment synthesis more clear; an overview of the entire process in order is provided as two flowcharts. The first for preparation and the second for performing the synthesis.





Step 3: Determine Quantities of Metal Oxides Needed

You can accomplish this task either by doing all the calculations, called stoichiometry, yourself or by using an Excel speadsheet I provide that will do the calculations. You may wish to use the spreadsheet if only to check your calculations. YInMn blue has a chemical formula of $Y_1In_{(1-x)}Mn_xO_3$. YInMn blue varies in darkness depending on how much manganese is present indicated in this formula by the variable "x". Standard YInMn blue has x=0.2 with the formula therefore being $Y_1In_{0.8}Mn_{0.2}O_3$. This means for every 10 atoms of yttrium, there are 8 of indium and 2 of manganese, Because we cannot count out individual atoms, chemists use a concept called the mole. It is a way of relating the atom counts in chemical substances to amounts of chemicals we can measure out. It is one the first concepts taught in chemistry, so chances are if you have any education in chemistry you already know it. Teaching in detail how the concept of the mole is used and how stoichiometry is done would be a whole additional instructable. And it already is one. And there are lots and lots and lots of online tutorials about these concepts. So I will only cover a very simple version of the idea here and those already familiar with it can skip the next two paragraphs.

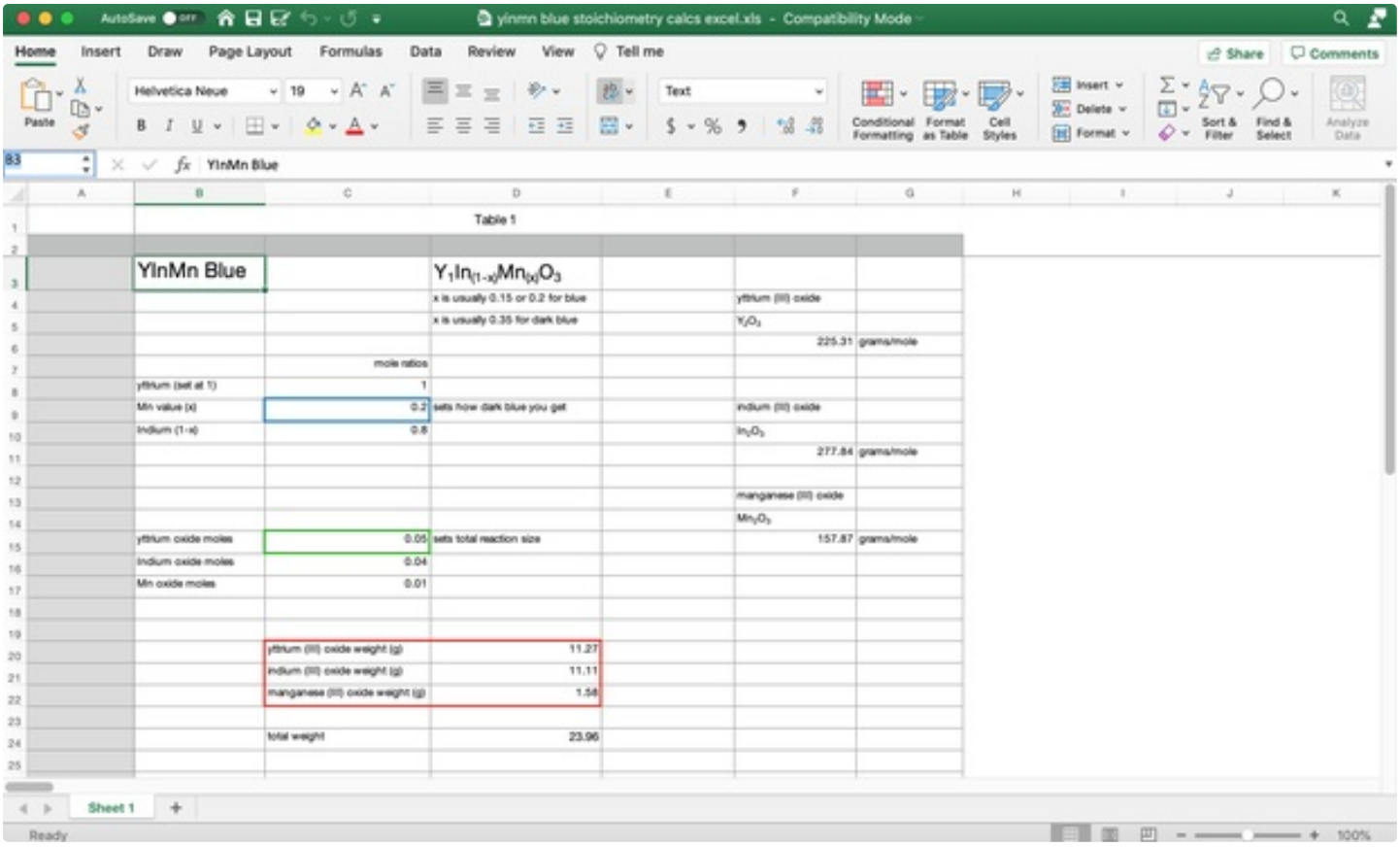
A mole (abbreviated "mol") of something is a bit like a dozen. Except that a dozen is 12 of an item, and mole is 6.02×10^{23} of it. So 1 mole of water molecules contains 6.02×10^{23} water molecules, just as a dozen water molecules would contains 12 of them. This means that a chemical equation applies equally well to moles of molecules as to individual molecules. Consider the chemical equation for burning hydrogen to make water: $2H_2 + O_2 \text{ ----> } 2H_2O$. It says that 2 hydrogen gas molecules (containing 2 hydrogen atoms each) react with one oxygen gas molecule (containing 2 oxygen atoms) to make 2 water molecules (containing 2 hydrogen atoms and one oxygen atom each). Which also means 2 moles of hydrogen gas (each containing 2 moles of hydrogen atoms) will react with one mole of oxygen gas (containing 2 moles of oxygen atoms) to make 2 moles of water (containing 2 moles hydrogen and one mole oxygen each).

So chemical equations that tell us how many atoms of each type are needed also tell us how many moles of each are needed. And that is important, because it is easy to relate a mole of a compound with a measurable weight of that substance. The weight of a mole of a molecule is equal to its molecular weight in grams and is often called the molar mass. The molecular weight is equal to the sum of the atomic weights of each atom in the molecule. In other words, add the atomic weight of every atom in the molecule together to find the molecular mass. Each atom has an atomic weight that can be looked up and is found on most periodic tables. For hydrogen it is 1.008 which is rounded to 1, for oxygen it is 15.999 which is rounded to 16 (the reason the weights are not simple integer amounts is because elements typically have several forms each with different numbers of neutrons and different abundances, the fractional weights account for these). So the molar mass of hydrogen gas is 2g/mol, for oxygen gas 32g/mol and for water 18g/mol. To make 36 grams of water, one needs 4 grams of hydrogen gas, and 32 grams of oxygen gas.

With these relationships in mind, it isn't all that hard to run the numbers yourself. The molar mass of yttrium oxide is 225.31 grams/mol, for indium oxide 277.84 grams/mol, and for manganese (III) oxide 157.87 grams/mol. Because all three have 2 metal atoms each we do not need to make any corrections as we would if they had different numbers of metal atoms. While one mole of the metal oxide contains 2 moles of metal atoms, this is true for all three and thus the 2 will cancel out.

The molar ratio (ratio of number of moles of each atom) needed for standard x=0.2 YInMn blue is one mole of yttrrium oxide to 0.8 moles of indium oxide to 0.2 moles of manganese (III) oxide. So if you want to make standard YInMn blue and want to react 1 mole of yttrium oxide (225.31 grams of it) you need 222.27g of indium oxide (277.84 g/mol multiplied by 0.8 mol = 222.27g) and 31.57 grams of manganese (III) oxide (157.87 g/mol multiplied by 0.2 mol = 31.57g). But that is a lot more than we probably want to make. A more reasonable size would use 0.03 moles of yttrium oxide. Multiply each result by 0.03 to get 6.76g of yttrium oxide, 6.67g of indium oxide, and 0.95 grams manganese (III) oxide. This makes about 14 grams of YInMn blue. To make YInMn dark blue, a navy blue-like color, use a yttrium to indium to manganese ratio of 1 : 0.65 : 0.35. For a sky blue like color use a 1 : 0.99 : 0.01 ratio.

While the calculations are straightforward, it is even easier to use the spreadsheet, at least to double check your results. If the concept of the mole was alien to you, this is probably the way to go. In the Excel spreadsheet I provide for these calculations, only alter the numbers in the blue and green lined boxes. You set the darkness of the pigment by changing the number in the blue lined box to the desired "x" value (0.01 for sky blue, 0.2 for standard YInMn blue, and 0.35 for dark blue) to set the manganese concentration. Change the number in the green lined box to set the number of moles of yttrium oxide used. This sets the overall reaction size, and determines the weight of each components needed as well as the total mass of pigment produced. I recommend 0.03, 0.05 or whatever is the lowest value that produces a value for the amount of manganese (III) oxide needed that is above 0.3 to 0.5 grams. The reason being that weight measurements smaller than this are hard to do consistently, especially for beginners. But feel free to make larger reactions to make more pigment, just remember that if something goes wrong you will be out larger amounts of the metal oxides. And indium oxide in particular is expensive. So I recommend keeping it small.



Step 4: Weigh Out and Prepare Metal Oxides

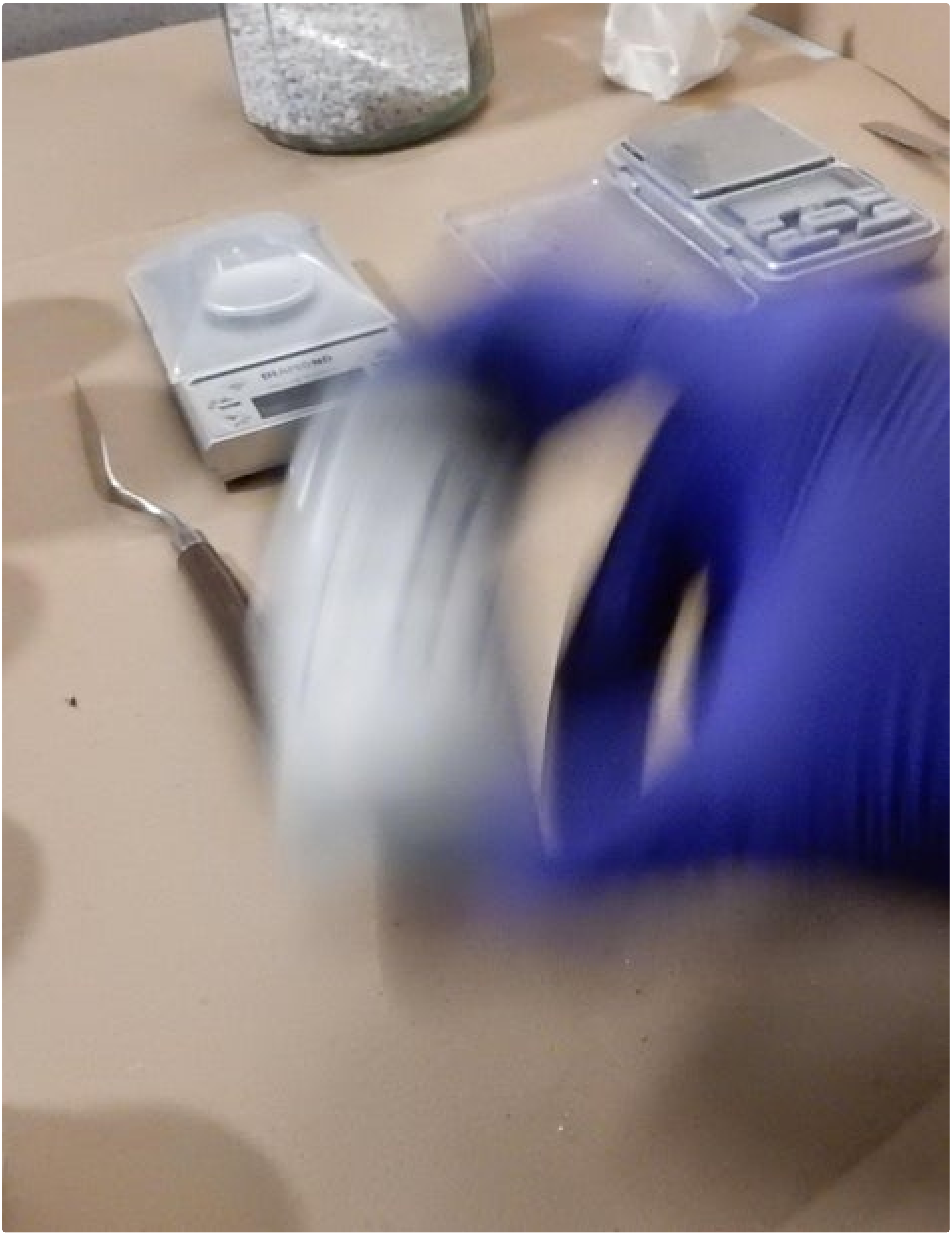
So now that you know how much of each oxide you need, now you have to weigh it all out. If you are not familiar with chemistry lab procedures, you may be wondering how are you going to weigh less than a gram of manganese (III) oxide. That is where the digital scale comes in and where I will refer to another instructable I wrote as a companion to this one. It describes how to weigh small amounts of chemicals (or most anything) and includes not only how to use a digital scale for this purpose, but also my suggestions on how to physically move around such tiny quantities. Find it here.

I strongly recommend using glassine papers for the manganese (III) oxide. Manganese (III) oxide will come cleanly off glassine papers, unlike many other weighing container materials. I also suggest that if you have one, use a digital scale that measures to the closest milligram (0.001 gram) for the manganese (III) oxide. This moves from recommended to nearly essential if you are trying to weigh out 0.3 grams or less and have any desire for your results to be consistent from experiment to experiment.

So if you followed the weighing tutorial or already know how to weigh out chemicals, you should have weigh boats or cups with yttrium oxide and indium oxide in them, and a glassine paper with manganese (III) oxide on it. Select a container that seals airtight holding at least 2-3 times more material than you have total metal oxides. An empty pill bottle works nicely Add the yttrium oxide first. I recommend doing this over a new CLEAN sheet of paper in case you spill any. If you do, the paper will catch it and because it is new and clean you can just add what spilled to the bottle. Then very carefully add the manganese (III) oxide by pouring it off the glassine paper on top of the yttrium oxide. You can try doing this over a clean glassine paper to recover any spilled, but try your best not to need it. Then finally, over a clean sheet of paper, add the indium oxide (if the paper from the yttrium oxide pour was not contaminated with anything but yttrium oxide, it can be reused). This is the most expensive component and I suggest adding it last. If you add it first and have anything go wrong except a minor recoverable spill, you may have to toss everything and start over. Adding it last

ensures that you loose the indium oxide only if you make a non-recoverable error transferring it, not two later additions as well. Adding the sticky manganese (III) oxide in the middle helps get it coated with the other oxides before it can contact the walls of the mixing bottle.

Once all three oxides are in, seal the mixing bottle and shake it like your life (or at least the money you spent on metal oxides) depends on it. You can't shake it too much. After at least 30 seconds of shaking, let the dust settle in the bottle (30 seconds should do it), then pour into a clean mortar. Avoid breathing the dust. Get the pestle, and grind up the mixed metal oxides. Do this in a circular motion, careful not to spill. As with transfer, a new clean sheet of paper underneath the mortar to catch spills is your friend here. Really grind this well. If you made your own manganese (III) oxide you will likely feel the larger particles as roughness when grinding, try to keep grinding until you don't feel the roughness any more. Grind at least 5 minutes or longer. The better ground this is, the easier time you will have regrinding in the next step. Time grinding here will save time down the road.



Step 5: Run the Reaction

Now that you have well ground mixed metal oxides, you need a crucible that is clean or has only been used for YInMn blue making. Crucibles used previously for making the pigment can be cleaned up a bit by rubbing the interior with a dry paper towel and removing any powder that loosens. If the crucible has been previously used only for making YInMn blue with the same concentration of manganese, cleaning is optional. Carefully transfer the ground powder into the crucible. As you might expect, having a new clean piece of paper underneath to catch spills helps here too. Use a transfer tool to scrape off the oxide mix sticking to the bottom and sides of the mortar. When you have gotten off as much as you can by scraping, take a new clean paper towel, bunch it up and lightly rub the bottom and sides of the mortar. Some of the oxides will stick to the paper towel, but more will collect as powder and can be transferred to the crucible.

Using the flat end of the pestle that is normally held, tamp down the powder in the crucible. Lightly at first, as otherwise the powder will fly everywhere. Once partially tamped down, you can do this harder. It is difficult to know how much tamping is enough, but my experience suggests that when tamping in one place starts to break up or move tamped

powder that is close to where the tamping is being done, you have done all you can do. Further tamping will not help with the reaction. You would need a pellet press to do more, and those can cost as much or more than your furnace.

Place the crucible into your furnace. Don't let the crucible touch the heating wire, but if running several crucibles, the crucibles can touch each other. If you have lids they can be used, but are not necessary unless you are trying to really pack the furnace and want to out a small crucible on top of a flat lid of a larger crucible. I've done it, this works just fine. Once your furnace is packed, close it, turn on main power and set the temperature for 1200°C (about 2200°F), Do not go lower. You will not get YInMn blue. You will get a paler, greyer blue down to about 1000°C and little or color change much below that. Let the furnace heat up, start the timer when it reaches 1200°C. Run 6 or more hours at 1200°C. Then turn off heat, or set the temperature on the furnace to below room temperature (I use anything under 10°C). And let it cool to room temperature. I like to set the temperature to below room temperature so that the PID controller on the furnace continues to report the temperature in the furnace.

When you open the furnace, your once grey oxide powder mix should be blue. A paler, less vivid blue than the final pigment, but it should be blue. If it isn't blue, either the temperature was not high enough, or one or more of the metal oxides was either not pure enough, was contaminated before reaching you or contaminated during your preparation. This is why the papers to catch spills were always new and clean. If there was contamination, the mix will likely be brownish, while if the temperature was simply too low, the powder will be grey without any hint of a brown tone.

The now blue heated oxide mix (called the pigment from here on) should be transferred to the cleaned mortar and pestle. It will be lightly sintered together. Running a thin tool between the pigment and the wall of the crucible may dislodge is all at once. Or you may need to use the tool to carefully break up chunks of the pigment, hopefully done without breaking the crucible. Once in the mortar, use the pestle to crush the chunks. You will see small white or pinkish white flecks inside the mass of pigment. The better you ground the pigment in the prior step the fewer and smaller they will be.

Grind the pigment up paying special attention to crushing and grinding as many of the flecks as possible. The flecks will be much harder than the rest of the pigment. Really grind them hard. When you have done as much as you think you can, try to grind a little more, then transfer it back to the crucible it was prepared in. Tamp the powder down again, reload the furnace and heat again just as before to 1200°C for 6 or more hours. Let the furnace cool. Then transfer the reheated pigment back to the mortar. Crush the pigment up again. You should see fewer, smaller flecks and the pigment will likely appear more vividly blue.

Regrind the pigment, again paying special attention to the flecks. Grind them down as much as you can. If you did a very thorough job with the initial grind of the oxide mix, there may be only a few flecks. If you did not grind well at this step you will be doing a lot more work here. Once again, grind them down as much as you can, transfer back to the crucible, tamp the powder down, and heat to 1200°C for 6 or more hours again. Let the furnace cool. The pigment should be a vibrant, brilliant blue under strong light and crushing it in the mortar and pestle should reveal no or at most rare tiny flecks visible only with a magnifying lens. Keep grinding your pigment. This time because well ground pigments work best for paint and pretty much any other use. Put your pigment in a clean, labeled container to store it. You can show it off and explain how you made the famous, but infamously hard to find, YInMn blue with just a little solid state chemistry. The pigment can be used to make many types of paint as well as to color castable resins.

Step 6: Troubleshooting

The most common problem you will face is that after 3 cycles of heating and regrinding, you still have white flecks. If you do, you will need more cycles of grinding and heating. Grind the flecks up and reheat. It may help to heat longer, 8-12 hours, especially if the flecks are small but abundant in number. Keep regrinding and reheating as long as with each cycle the flecks diminish. If they do not, you may need to try another batch.

If you have trouble physically using a mortar and pestle, I have had good results using a ball mill made from a harborfreight rock tumbler. However ball milling is a lossy process with small samples due to the high fixed mass retained by the apparatus. Stainless steel balls in a stainless steel jar worked best for me, although I have used alumina ceramic in high density polyethylene jars between firings. Any polyethylene scraped off into the pigment will be incinerated into carbon dioxide and water during heating to 1200°C.

If the pigment looks greyish, even with reheating, your furnace is probably not hot enough. Recalibrate your

thermocouple, or get a new one if it is old or the wires have started to thin. Make sure you are using the correct type of thermocouple. Type K is most common. However, 1200°C is at the upper end of its range (-100°C to +1250°C) and is very hard on the thermocouple wires reducing its life considerably. A type K used in kilns that has ceramic pieces for physical protection and a bare wire tip is usually the least expensive one that will work. Ones with a protective stainless steel sheath will not tolerate 1200°C, though sellers sometimes make this claim (reviews usually say otherwise). A type R or S will be more accurate and last longer but will be much more expensive (they use platinum wires). If you change thermocouple type, remember to update the PID controller settings to match.

If your furnace seems to be able to heat above 1200°C you can try that. Temperatures above 1200°C will, if anything, do a better job in this synthesis. And if your thermocouple is reading high, what you read as 1300°C may actually be 1200°C. Keep in mind that Kanthal A1 wire cannot heat above about 1400°C and nichrome 80 much above 1200°C. If you go higher they may melt and even below those maximum temperatures will sag requiring more support pins.

If your pigment is brown, it is likely due to either contaminating the oxide mix during preparation or using metal oxides that are not pure enough or were adulterated. If you did not spill and scrape up your mix, used clean transfer tools, and employed fresh crucibles, it is almost certainly the metal oxides as sourced that were the problem. Try different sources or seek reliable advice on technique and material sourcing.

Step 7: References and Further Directions

Scientific journal articles relating to the development and characterization of YInMn blue and used to create this

protocol, can be found at a google drive I have set up to provide access to Excel spreadsheets for stoichiometry calculations. These include papers and Excel spreadsheets useful in making green and purple color variants (shown above) by substituting aluminium, titanium, copper and zinc oxides for some of the indium/manganese in the basic YInMn blue synthesis. Yellow, orange, and brick red have also been reported but I have not yet been successful in making them perhaps due to not being able to heat above 1200°C. You can find additional information about these on the subreddit DIYPigments where I am a moderator. I have also included several pdf references relating to the making of paint that may also be useful. Please feel free to contact me here with any questions and I will try to help.

Step 8: Acknowledgements

I would like to thank reddit user formallydehyde for their initial reports trying to make YInMn blue at home, braving the dangers of the antique dental furnace "Ol' Terrifying." Their work made me realize it was realistic to try to make YInMn blue at home, laying the groundwork for success. And of course, special thanks to Dr. Mas Subramanian and Dr. Andrew Smith, as well as others in the Subramanian lab without whose work and insight the world would likely still be unaware of this new chromophore. Chance does indeed favor the prepared mind.


Excellent, thank you!


You're very welcome and thanks for looking past the typos in the initial publication!


Interesting! Thanks for sharing :)

You're welcome! Unfortunately I almost didn't get it in on time and a lot of typos didn't get cleared. And there were a lot of them! Combination of arthritic thumbs and a brain with a propensity when proofreading too soon after writing to overlook typos and see what I meant to write not what I wrote.

But it looks like I can start getting these cleaned up even while it is in judging, as the judged version doesn't change. So I started on that today.

While the companion instructable on weighing for chemistry was technically my first submission (by about 1 hour), it was created as part of this project. So this is pretty much my first submission. There is a lot of room for improvement, especially on images and layout.